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COMPLETE SPECIFICATION

New Plastisols and Organosols

5 We, CIBA LIMITED, a Swiss Body Corporate, of Basle, Switzerland, do hereby declare the invention for which we pray a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 It is known that objects, more especially those made from iron or non-ferrous metals, can be coated by treating them with a so-called plastisol or organosol containing a powdered vinyl resin dispersed in a suitable plasticizer, whereupon the resulting coating is heated to a suitable gelling temperature.
15 For this method there are suitable numerous polyvinyl resins, especially polyvinylchloride and copolymers of vinylchloride with other unsaturated monomers. In this manner there are obtained tough and non-abrading coatings
20 which, however, have the unfavourable property that they adhere very poorly to most surfaces.

To remedy the said disadvantage it has already been proposed to replace the conventional
25 plasticizers, e.g. dioctyl phthalate, either wholly or partially by a polymerizable plasticizer, such as diallyl phthalate, and further to add a polymerization catalyst, for example an organic peroxide. During gelling the plasticizer is at the same time polymerized; in
30 general, this procedure results in an improvement only if at the same time certain lead compounds, such as white lead, dibasic lead phthalate or dibasic lead carbonate are further

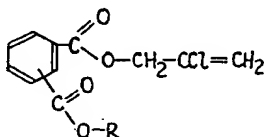
added. However, in certain spheres of application the use of poisonous lead compounds is inadmissible. In addition, the coatings produced in this manner often display insufficient stability to light.

The present invention is based on the unexpected observation that even without adding such lead compounds coatings and cemented products having outstandingly good adhesivity, especially on iron and non-ferrous metal surfaces, can be obtained by using as
45 polymerizable plasticizer a 2-chlorallyl phthalate instead of diallyl phthalate. By adding a basic compound, e.g. calcium hydroxide, the adhesion can be further enhanced.

It is specially surprising that it is possible
50 to obtain in this manner coatings and cemented products having an optimum adhesivity which is much superior to the best adhesivity achieved with the known plastisols based on polyvinylchloride, diallyl phthalate
55 and the afore-mentioned lead compounds.

Accordingly, the present invention provides plastisols and organosols which are particularly suitable for cementing and coating iron and non-ferrous metals, and contain a pulverulent, halogen-containing vinyl polymer, a
60 polymerizable plasticizer in which the said polymer is substantially insoluble at room temperature, a polymerization catalyst, as well as a stabilizer for the vinyl polymer, wherein the plasticizer consists at least partially of a compound of the formula
65

(I)



where R represents an aliphatic, cycloaliphatic, araliphatic or aromatic residue,

preferably a 2-chloroallyl residue, an allyl residue or an aliphatic hydrocarbon residue containing 4 to 18 carbon atoms.

5 The plasticizer of the formula (I) to be used in this invention is a diester of terephthalic, isophthalic or especially orthophthalic acid in which at least one of the two carboxyl groups is esterified with 2-chloroallyl alcohol. The other carboxyl group is preferably likewise esterified with 2-chloroallyl alcohol, though other esterifying alcohols, such as an allyl alcohol, methanol, ethanol, propanol, isopropanol, n-butanol, isobutanol, tertiary butanol, an amyl alcohol, a hexanol, n-octyl alcohol, 2-ethyl-hexanol-1, lauryl alcohol, stearyl alcohol, oleyl alcohol, cyclohexanol or benzyl alcohol are equally suitable.

20 As examples of suitable polymerizable plasticizers of the formula (I) there may be mentioned: bis(2-chloroallyl)isophthalate, bis(2-chloroallyl)terephthalate, allyl-2-chloroallyl-ortho-phthalate, 2-ethylhexyl-2-chloroallyl-ortho-phthalate and especially bis(2-chloroallyl)-ortho-phthalate.

25 The plasticizers of the formula (I) may be used by themselves or in conjunction or admixture with other known polymerizable or unpolymerizable plasticizers for polyvinylchloride, e.g. phthalates such as dioctyl phthalate, diallyl phthalate, sebacates such as dibutyl sebacate, or adipates such as dibutyl adipate.

As polymerization catalysts there are gen-

erally used the known catalysts capable of furnishing free radicals. There may be mentioned hydrazine derivatives, e.g. hydrazine hydrochloride; organometal compounds such as tetraethyl lead, and especially aliphatic azo compounds such as α, α' -azoisobutyronitrile and organic peroxides or persalts, such, for example, as peracetic acid, acetyl peroxide, chloroacetyl peroxide, trichloroacetyl peroxide, benzoyl peroxide, chlorobenzoyl peroxide, benzoylacetyl peroxide, propionyl peroxide, fluorochloropropionyl peroxide, lauryl peroxide, cumene hydroperoxide, tertiary butyl hydroperoxide, di-tertiary butyl peroxide, di-tertiary-amyl peroxide, *para*-menthane hydroperoxide and tertiary butyl perbenzoate.

As pulverulent vinyl polymers to be used in the plastisols or organosols of this invention there may be mentioned: Polyvinylchloride, copolymers of vinylchloride with other unsaturated monomers, such as vinyl acetate, maleic anhydride, ethylene, acrylonitrile, vinylidene chloride and acrylic acid esters. Furthermore, there may be used polymers that have been after-treated, such as chlorinated or hydrolyzed, e.g. chlorinated polyvinyl chloride or partially hydrolyzed copolymers of vinyl acetate or vinyl acetals with vinylchloride. There may be used a pulverulent, commercial polyvinylchloride as is conventionally used for making pastes, such as has been described in *Plastics Engineering Handbook* (Soc. Plast. Ind.) Reinhold Publishing Corp., New York [1954] pages 270 et seq. Suitable types are marketed, for example, under the registered trade marks GEON 121, GEON 202, VINYLITE QXNV, MARVINOL VR-10, VESTOLIT G, VESTOLIT PH, OPALON 410, PLIOVIC AO and DOW X 2702. Equally suitable are commercial polyvinyl pastes that as such already contain proportions of plasticizer and stabilizer, for example the product marketed by I.C.I. under the registered trade mark WELVIC Paste, having the following composition:

VINYLCHLORIDE POLYMER P 65/54 (registered trade mark)	50 parts
VINYLCHLORIDE POLYMER D 55/3 (registered trade mark)	50 „
PLIABRAC 980 (registered trade mark for an adipic acid ester plasticizer)	25 „
LUBROL MOA (registered trade mark for a polyethylene oxide viscosity reducer)	2 „
ABRAC A (registered trade mark for an epoxidized soybean oil)	2 „
STANCLERE 80 (registered trade mark for an organotin stabilizer)	0.75 part

The plastisols or organosols of this invention further contain the usual heat and light stabilizers. There may be mentioned metal salts of higher fatty acids, such as calcium stearate, calcium palmitate, calcium oleate, calcium ricinoleate, strontium stearate, barium stearate, barium laurate, cadmium laurate and lead stearate; mixed metal soaps such as barium-cadmium palmitate and barium-cadmium laurate; a few other barium and cadmium compounds, especially basic salts, such as dibasic leadphosphite

($2\text{PbO} \cdot \text{PbHPO}_3 \cdot \frac{1}{2}\text{H}_2\text{O}$),
dibasic lead phthalate, basic lead stearate and dibasic lead carbonate; organotin compounds, such as dibutyl tin di-fatty acid salts, e.g. dibutyl tin dilaurate, dibutyl tin dioleate or dibutyl tin diricinoleate, dibutyl tin maleate and di-n-octyl tin maleate; di-butyl-tin-dithioglycollic acid esters such as di-butyl tin diisooctyl thioglycollate; furthermore, monoepoxy and polyepoxy compounds likewise act as stabilizers.

As monoepoxides there may be mentioned: butyl glycidyl ether, allyl glycidyl ether, phenylglycidyl ether, cresylglycidyl ether, styrene oxide, 3,4 - epoxytetrahydrodicyclopentadienol - 8, 3,4 - epoxyhexahydrobenzal glycerol, cyclohexene oxide, 3,4 - epoxycyclohexane - 1,1 - dimethanol acrolein acetal, acrylic acid glycidyl ester and tetrahydrofurfuryl glycidyl ether.

As polyepoxides there may be mentioned: Alicyclic polyepoxides such as vinyl cyclohexene dioxide, limonene dioxide, dicyclopentadiene dioxide, ethyleneglycol - bis(3,4 - epoxy - tetrahydrodicyclopentadien - 8 - yl) - ether, (3,4 - epoxy - tetrahydrodicyclopentadien - 8 - yl) - glycidyl ether; epoxidized

polybutadienes or copolymers of butadiene with ethylenically unsaturated compounds, such as styrene or vinyl acetate; compounds containing two epoxycyclohexyl residues, such as diethyleneglycol bis - (3,4 - epoxycyclohexane - carboxylate), bis - (3,4 - epoxycyclohexylmethyl) - succinate, 3,4 - epoxy-6-methylcyclohexylmethyl - 3,4 - epoxy - 6 - methylcyclohexane carboxylate and 3,4 - epoxyhexahydrobenzal - 3,4 - epoxycyclohexane - 1,1 - dimethanol. Polyglycidyl esters, such as are accessible by reacting a dicarboxylic acid with epichlorohydrin or dichlorohydrin in the presence of alkali, for example diglycidyl adipate and diglycidyl phthalate.

Polyglycidyl ethers, such as are accessible by etherifying a dihydric or polyhydric alcohol, or diphenol or polyphenol, with epichlorohydrin or dichlorohydrin in the presence of alkali. These compounds may be derived from glycols such as ethyleneglycol, 1,4 - butyleneglycol, or from diphenols or polyphenols such as resorcinol or phenol-formaldehyde condensation products of the resol or novolak type; bis - (para-hydroxyphenyl)-methane and especially bis - (para-hydroxyphenyl)-dimethylmethane.

It is, of course, also possible to use a mixture of two or more of the above-mentioned monoepoxides and/or polyepoxides.

In many cases a particularly good stabilizing effect is obtained with mixtures of two or more of the above types of stabilizers.

In addition, there may be further used other types of stabilizers, especially light stabilizers, e.g. certain organic pigments and ultraviolet absorbers.

The adhesion of the resulting coatings or cementations can be further increased by

adding basic substances to the organosols or plastisols of this invention, such as the basic stabilizers referred to above, for example basic lead compounds. It is, however, preferable to add basic compounds not acting as stabilizers in combination with nontoxic stabilizers of neutral reaction, such as organotin compounds or barium-cadmium stabilizers. As such preferred basic additives there may be mentioned in the first place alkaline earth metal hydroxides, such as calcium hydroxide and barium hydroxide.

For the manufacture of organosols there are further added organic solvents or swelling agents, e.g. acetone, tetrahydrofuran, cyclohexanone, toluene or xylene.

The plastisols or organosols of this invention may further contain mineral fillers, such as titanium dioxide, iron oxide, vegetable fillers, reinforcing agents, pigments and dye-stuffs.

The plastisols or organosols are manufactured in the usual manner, for example by mixing the components at room temperature in a suitable mixing apparatus, such as a ball-mill, three-roller mill or the like, making sure that the temperature does not rise above 30°C.

The plastisols and organosols of this invention are suitable for the manufacture of coatings on iron metals such as mild steel, non-ferrous metals such as copper or brass, also on wood, ceramics or textiles of polyamide or polyester fibres, or as adhesives for cementing together two surfaces of the materials just mentioned. They may also be

used for the manufacture of laminates, castings, printing inks, and as putties, dipping resins or potting compounds. By addition of propellants, it is further possible to manufacture foamed products.

Percentages in the following Examples are by weight.

EXAMPLE 1

Cements for metal sheets

Six cementing compounds are manufactured by mixing polyvinylchloride (paste grade; registered trade mark VINNOL P 100/70; makers Wacker-Chemie GmbH, Munich) with bis(2 - chlorallyl phthalate) as polymerizable plasticizer, dioctyl phthalate as extender, tertiary butyl perbenzoate as catalyst, and various lead, barium-cadmium and organotin stabilizers in the proportions shown in the following Table 1.

One test series each was carried out with and without addition of calcium hydroxide.

The ingredients of each compound are thoroughly mixed and ground twice on a three-roller mill. For cementing purposes the resulting pastes are applied in a layer about 0.1 mm. thick on iron strips (pickled once) measuring 170×25×1 mm.; the iron strips are fixed with a 15 mm. overlap and each cemented strip was allowed to gel for 90 minutes at 170° C, whereupon the shear strength of each cemented specimen was measured. The values thus obtained are a measure of the adhesion of the gelled cement on the metal base. The results achieved are summarized in the following Table:

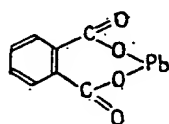
TABLE 1

	Grams of Cement					
	1	2	3	4	5	6
VINNOL P 100/70 ¹⁾	100	100	100	100	100	100
bis(2-chlorallyl)phthalate	38.4	38.4	38.4	38.4	38.4	38.4
di-n-octylphthalate	30	30	30	30	30	30
titanium dioxide (rutile)	10	10	10	10	10	10
tertiary butyl perbenzoate	0.6	0.6	0.6	0.6	0.6	0.6
basic lead carbonate ²⁾	10	10	—	—	—	—
dibasic lead phthalate ³⁾	10	10	—	—	—	—
barium-cadmium stabilizer ⁴⁾	—	—	4	4	—	—
organotin stabilizer ⁵⁾	—	—	—	—	4	4
calcium hydroxide	—	4	—	4	—	4
Shear strength kg/mm ²	1.83	1.98	1.18	1.85	1.55	1.90

¹⁾ Polyvinylchloride (makers Wacker-Chemie GmbH)

²⁾ $2 \text{ PbCO}_3 \cdot \text{Pb(OH)}_2$

³⁾



⁴⁾ Barium-cadmium salt of palmitic acid (registered trade mark STABILISATOR ZPS, makers Baerlocher, Munich)

⁵⁾ Modified dibutyl tin maleate (registered trade mark ADVASTAB 5216, makers Deutsche Advance Prod., Marienberg)

Similar results are obtained by cementing strips of copper, brass or stainless steel (V-4 A) sheet instead of iron strips.

5 For comparison with the cements 1 to 6 of this invention cements 7 to 12 of identical compositions were prepared in which, however, bis(2 - chlorallyl phthalate) was

replaced by an equivalent quantity of diallyl phthalate.

Cements 7 to 12 were used for cementing together iron strips as described for cements 1 to 6 and then gelled for 90 minutes at 170° C. The compositions of the cements and the shear strength values of the cemented strips obtained are shown in the following

10

15

TABLE 2

	Grams of Cement					
	7	8	9	10	11	12
VINNOL P 100/70	100	100	100	100	100	100
diallyl phthalate	30	30	30	30	30	30
di-n-octylphthalate	30	30	30	30	30	30
titanium dioxide (rutile)	10	10	10	10	10	10
tertiary butylperbenzoate	0.6	0.6	0.6	0.6	0.6	0.6
basic lead carbonate	10	10	—	—	—	—
dibasic lead phthalate	10	10	—	—	—	—
barium-cadmium stabilizer	—	—	4	4	—	—
organotin stabilizer	—	—	—	—	4	4
calcium hydroxide	—	4	—	4	—	4
Shear strength kg/mm ²	1.36	1.50	0.37	0.83	0.44	0.89

- 5 Finally, for further comparison with the cements of this invention similarly composed cements 13 to 18 were manufactured in which, however, bis(2 - chloroallyl)phthalate was completely replaced by di - n - octylphthalate. Iron strips were cemented together with these cements as had been done with cements 1 to 6, and then gelled for 90 minutes at 170° C. The compositions of the cements 13 to 18 and the shear strength values of the cemented strips are shown in the following 10

TABLE 3

	Grams of Cement					
	13	14	15	16	17	18
VINNOL P 100/70	100	100	100	100	100	100
di-n-octylphthalate	60	60	60	60	60	60
titanium dioxide (rutile)	10	10	10	10	10	10
tertiary butylperbenzoate	—	—	—	—	—	—
basic lead carbonate	10	10	—	—	—	—
dibasic lead phthalate	10	10	—	—	—	—
barium-cadmium stabilizer	—	—	4	4	—	—
organotin stabilizer	—	—	—	—	4	4
calcium hydroxide	—	4	—	4	—	4
Shear strength kg/mm ²	0.64	0.71	0.25	0.49	0.49	0.25

The cements 1—18 may also be used for the manufacture of tough and non-abrading coatings on sheets of iron, copper or brass. The coatings obtained with the cements 1 to 6 of this invention display in all cases better adhesion than the coatings produced with the cements 7 to 12 and 13 to 18, using identical stabilizer systems.

EXAMPLE 2

Cements 19 to 23 are manufactured as described for cements 1 to 18 in Example 1. The cements 19 to 23 were used for cementing together metal sheets under different gelling conditions, the stoving time and temperature used in each case being shown in Table 4. The cementing of the iron sheets was carried out as described in Example 1, except that an overlap of 10 mm was used.

TABLE 4

	grams of cement				
	19	20	21	22	23
VINNOL P 100/70 ¹⁾	100	100	100	—	—
LONZA D 61 ²⁾	—	—	—	100	100
bis(2-chlorallyl)-phthalate	48	48	48	48	48
(2-ethyl-n-hexyl)-(2-chlorallyl)phthalate	—	12	24	12.5	10
di-(2-ethyl-n-hexyl)-phthalate	30	12	—	12.5	10
titanium dioxide (rutile)	17.5	14	14	25	20
tertiary butylperbenzoate	0.75	0.6	0.6	0.5	2
barium-cadmium stabilizer ³⁾	5	4	4	—	—
ARALDITE F (reg. trade mark) ⁴⁾	—	—	—	3.75	3
calcium hydroxide	5	4	4	5	4
Shear strength kg/mm ²	2.30	2.50	2.45	2.22	2.24
Gelling conditions:					
minutes	50	30	30	25	25
° C.	180	180	180	175	175

¹⁾ Polyvinylchloride (makers Wacker-Chemie GmbH)

²⁾ Polyvinylchloride (makers Lonza A.G., Basle)

³⁾ Barium-cadmium salt of palmitic acid (registered trade mark STABILISATOR ZPS, makers Baerlocher, Munich)

⁴⁾ Liquid epoxy resin obtained by reacting epichlorohydrin with bisphenol A in the presence of alkali; contains 5.2 epoxide equivalents per kg; makers Ciba A.G., Basle)

EXAMPLE 3

Compounds for coating metal sheets

The plastisol is manufactured as described for the cements 1 to 18 in Example 1.

Composition of the plastisol

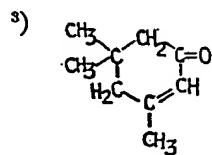
LONZA D 61 ¹⁾	100 grams
bis(2-chlorallyl)phthalate	50 „
(2-ethyl-n-hexyl) (2-chlorallyl)phthalate	30 „
di-2-ethyl-n-hexylphthalate	16.7 „
chrome oxide green	33.3 „
tertiary butylperbenzoate	2.7 „
barium-cadmium stabilizer ²⁾	5.0 „
calcium hydroxide	6.7 „

Composition of diluent

Isophorone ³⁾	16.2 grams
toluene	8.1 „

¹⁾ Polyvinylchloride (makers Lonza A.G., Basle)

²⁾ Barium-cadmium salt of palmitic acid (registered trade mark STABILISATOR ZPS, makers Baerlocher, Munich)



To obtain it in a spray consistency, the plastisol must be mixed, for example, with the quantity of diluent shown above.

10 The diluted plastisol (= organosol) lends itself well to spraying with a "De Vilbiss" (Registered Trade Mark) Spraygun Model GD (pressure container gun, 1mm nozzle, compressed air of 2 atmospheres).

The spray should be adjusted so that the gelled film is 180 to 220 μ thick.

15

The following stoving conditions have been found the most favourable for metal strips (pickled once, 1mm thick, 170 \times 25mm), using a drying cabinet with circulating air:

20

20 to 30 minutes at 170°C.
15 to 25 „ „ 175°C.
10 to 15 „ „ 180°C.

EXAMPLE 4

Compound for coating sheet metal

25 Manufacturing the plastisol: All ingredients except the polyvinylchloride and peroxide are mixed in a water bath at 50 to 60°C, with the isophthalic acid ester turning liquid. The polyvinylchloride is added at room tempera-

ture. The mixture is ground twice on a three-roller mixer, whereupon the indicated amount of tertiary butylperbenzoate is added and the whole is intimately mixed.

30

The other test conditions, such as addition of the diluent, processing, film thickness, are the same as in Example 3.

35

Composition of the plastisol

LONZA D 61 ¹⁾	100 grams
bis(2-chlorallyl)isophthalate	50 „
(2-ethyl-n-hexyl) (2-chlorallyl) phthalate	40 „
di(2-ethyl-n-hexyl)phthalate	20 „
chrome oxide green	16 „
tertiary butylperbenzoate	2.6 „
barium-cadmium stabilizer ²⁾	5.0 „
calcium hydroxide	6.6 „

Composition of the diluent

butanol	4.4 grams
amyl alcohol	2.3 „
decalin	2.3 „

¹⁾ Polyvinylchloride (makers Lonza A.G., Basle)

²⁾ Barium-cadmium salt of palmitic acid (registered trade mark STABILISATOR ZPS, makers Baerlocher, Munich)

The following stoving conditions have proved most advantageous for metal strips (pickled once, 1mm thick, 170×25mm) using
5 a drying cabinet with circulating air:

20 to 30 minutes at 170°C.
15 to 25 „ „ 175°C.
10 to 15 „ „ 180°C.

EXAMPLE 5

Cement for sheet metal

The plastisol is manufactured in the way described in Example 4 for the metal coating compound, and the strips are cemented as described in Example 1.

10

Composition of the plastisol

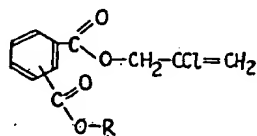
LONZA D 61 ¹⁾	100 grams
bis(2-chlorallyl) isophthalate	40 „
(2-ethyl-n-hexyl) (2-chlorallyl) phthalate	23.5 „
di(2-ethyl-n-hexyl)phthalate	11.5 „
chrome oxide green	13.5 „
tertiary butylperbenzoate	1.0 „
barium-cadmium stabiliser ²⁾	2.5 „
calcium hydroxide	3.3 „
Shear strength kg/mm ²	2.45
Gelling for 30 minutes at 170° C.	

¹⁾ Polyvinylchloride (makers Lonza A.G., Basle)

²⁾ Barium-cadmium salt of palmitic acid (registered trade mark STABILISATOR ZPS, makers Baerlocher, Munich)

WHAT WE CLAIM IS:—

1. Plastisols and organosols, particularly suitable for coating and cementing iron and nonferrous metals, containing a pulverulent, halogen-containing vinyl polymer, a polymerizable plasticizer in which the said polymer is substantially insoluble at room temperature, a polymerization catalyst and furthermore a stabilizer for the vinyl polymer, wherein the plasticizer consists at least partially of a compound of the formula



where R represents an aliphatic, cycloaliphatic, araliphatic or aromatic residue.

2. Plastisols and organosols as claimed in Claim 1, wherein the polymerizable plasticizer is bis(2-chlorallyl)phthalate.

3. Plastisols and organosols as claimed in Claim 1, wherein the polymerizable plasticizer is (2-ethyl-n-hexyl)(2-chlorallyl)phthalate.

4. Plastisols and organosols as claimed in Claim 1, wherein the polymerizable plasticizer is bis(2-chlorallyl)isophthalate.

5. Plastisols and organosols as claimed in Claim 1, 2, 3 or 4, that further contain an unpolymerizable plasticizer.

6. Plastisols and organosols as claimed in any one of Claims 1 to 5, containing a substance of basic reaction.

7. Plastisols and organosols as claimed in Claim 6, wherein the substance of basic reaction is calcium hydroxide.

8. Plastisols and organosols as claimed in any one of Claims 1 to 7, wherein the halogen-containing vinyl polymer is a polymer or copolymer of vinylchloride.

9. Plastisols and organosols as claimed in claim 1 substantially as described in the Examples.

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